

SPECTROPHOTOMETRIC ANALYSIS No. A243

Auto Analysis for Mg & Mn in Metals by AA-6500 with Flame Micro Sampling (Single Drop Sampling)

Measurement with the flame micro sampling method requires only a very small volume of sample (about 50 to 100 μl) per measurement as compared with the ordinary flame method. Even a sample solution of high concentration or a sample with a high salt concentration can be measured with a small injection volume, without clogging of the burner.

By combining the new AA-6501 atomic absorption spectrophotometer with the ASC-6000 auto sampler,

■ Measurement of Magnesium in Aluminum Alloy

Weigh 0.5g of sample, pretreat according to JIS H 1357, and make up to 100 m^3 with water. Using the ASC-6000, add 10 ppm of magnesium, 10000 ppm of strontium, purified water, and 5000 ppm of aluminum.

single drop sampling may be realized. This system possesses an automatic sample preparation function, and the calibrating solution may be prepared by varying the sampling volume of a standard solution. The sample may be diluted, or an interference removing agent (e.g., Sr) may be added, so that automatic measurement is possible. Examples of measurement with single drop sampling are introduced below.

Then add reagents, including automatic dilution, in accordance with the reagent preparation program in Fig. 3, then analyze.

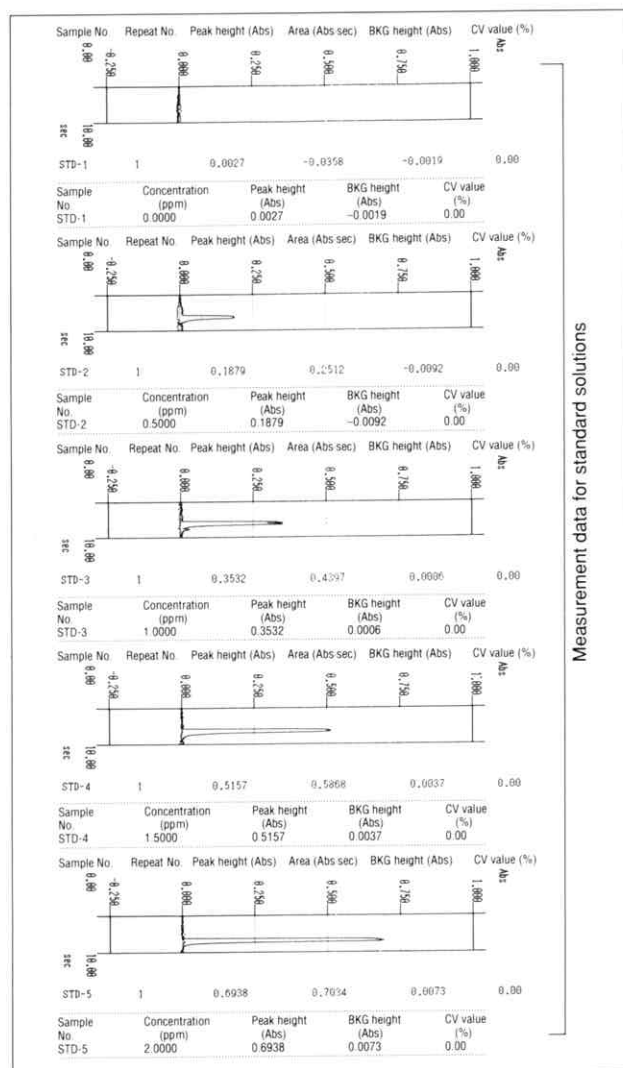


Fig. 1 Measurement of Mg Standard Solutions

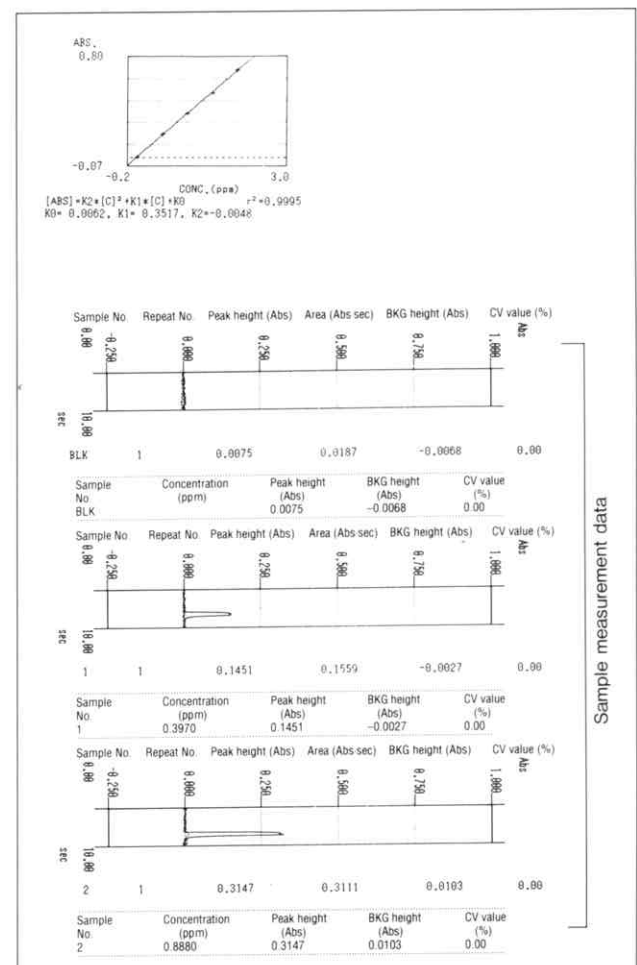


Fig. 2 Calibration Curve for Mg Standard Solution and Sample

**** Flame quantitative measurement (single drop method) parameters **** 1991/12/27 12:53:29 ****

Lighting conditions:	Lighting	Mn #2					
	Current	8 mA/0 mA					
	Wavelength	285.2 nm	Burner height	6 mm			
	Slit width	0.5 nm	Fuel gas flow rate	2.0 l/min			
	Lighting mode	BGC-02	Additive gas type	air			
	Analyzed by						
	Remarks						
Measurement parameters:							
Method of concentration calculation	Calibration curve method	No. of standard sample	5				
Signal processing mode	Peak height	Degree of calibration curve formula	2 nd				
Data sampling time	10 seconds	Zero point passing of calibration curve	NO				
Waveform display response	DIRECT	Unit of concentration	ppm				
Repeat conditions							
Blank measurement	1 times	Max. repeats	CV value limit				
Standard sample measurement (standard addition method)	1 times	1 times	99.9%				
Unknown sample measurement (simplified standard addition method)	1	1	99.9				
Drift correction measurement	1	1	99.9				
ASC parameters							
Automatic dilution re-measurement	OFF						
Blank Measurement alternative	OFF						
1) First reagent position	R1						
2) Second reagent position	R2						
3) Third reagent position	R3						
4) Fourth reagent position							
Mixing	ON						
Blended sample injection volume	70 µl						
Inspection speed	25 µl/sec						
Suction discharge time	5 times						
Suction speed	130 µl/sec						
Discharge speed	330 µl/sec						
STD							
Concentration	Standard sample position	Sample amount	R1 amount	R2 amount	R3 amount	Total	
No	ppm	R1 to R8/1 to 60	(µl)	(µl)	(µl)	(µl)	
1	0.0000	1	0	400	50	500	
2	0.5000	1	50	350	50	500	
3	1.0000	1	100	300	50	500	
4	1.5000	1	150	250	50	500	
5	2.0000	1	200	200	50	500	
		Sample position	Sample amount	R1 amount	R2 amount	R3 amount	Total
		R1 to R8/1 to 60	(µl)	(µl)	(µl)	(µl)	(µl)
Blank		R1	0	450	50	0	500
Drift							****
Unknown sample			50	400	50	0	500

Fig. 3 Conditions for Mg

***** Flame quantitative measurement (single drop method) ***** 1991/11/25 19:16:32 ***

Element name	Mn
Wavelength	279.5 nm
Method of concentration calculation	Calibration curve method
Analyzed by	
Remarks	

SAMPLE NO.	SAMPLE NAME	CONC. (ppm)	ABS. (285.2nm)	WEIGHT (g)	VOLUME (ml)	DILUTION RATIO	ACTUAL CONC.	FACTOR
1	No.1	0.3978	0.1451	0.508	100.00	10.00	0.079 (x)	1.00
2	No.2	0.8880	0.3147	0.502	100.00	10.00	0.177 (x)	1.00

Fig. 4 Analytical Results for Mg

The final analytical result is automatically calculated by entering the sample weight and dilution factor, to the value automatically calculated from the calibration curve.

Measurement for Manganese in Iron and Steel

Weigh 1g of sample, pretreat according to JIS G 1257, and make up 100 ml. Using the ASC-6000, and 5 ppm of manganese, purified water and 10000 ppm of iron. Then add the reagents, including automatic dilution, according

to the reagent preparation program in Fig. 6, and analyze. Here, omitting the measurement data for the standard solution, the data after the calibration are shown.

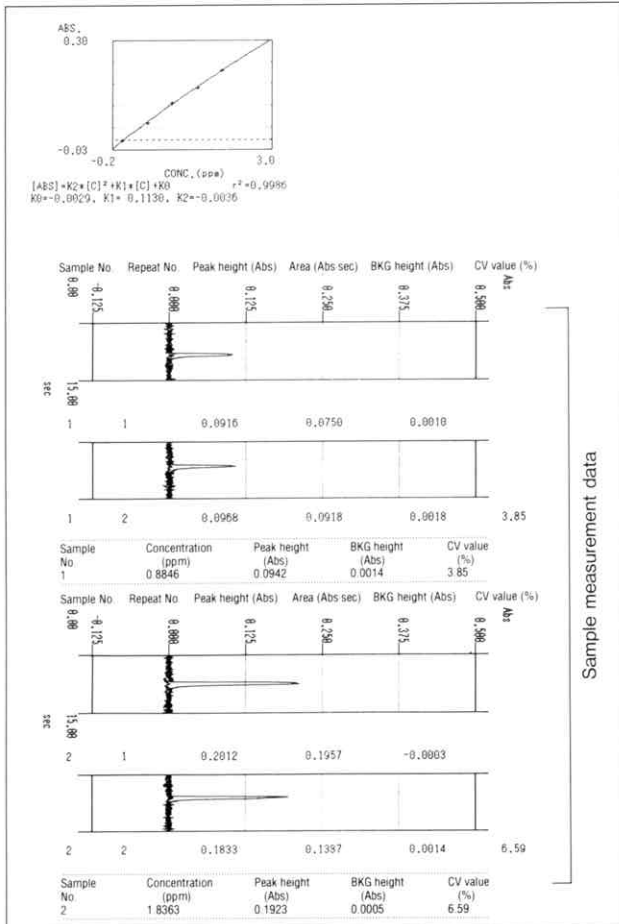


Fig. 5 Calibration Curve for Mn Standard Solution and Sample Measurement

**** Flame quantitative measurement (single drop method) parameters **** 1991/11/25 19:08:53 ****

Lighting conditions:	Lighting	Mn #2				
	Current	10 mA/0 mA				
	Wavelength	279.5 nm	Burner height	6 mm		
	Slit width	0.2 nm	Fuel gas flow rate	2.0 l/min		
	Lighting mode	BGC-02	Additive gas type	air		
	Analyzed by					
	Remarks					
Measurement parameters:						
Method of concentration calculation	Calibration curve method	No. of standard sample	5			
Signal processing mode	Peak height	Degree of calibration curve formula	2 nd			
Data sampling time	15 seconds	Zero point passing of calibration curve	NO			
Waveform display response	DIRECT	Unit of concentration	ppm			
Repeat conditions						
Blank measurement	1 times	Max. repeats	CV value limit			
Standard sample measurement (standard addition method)	2	2	99.9			
Unknown sample measurement (simplified standard addition method)	2	2	99.9			
Drift correction measurement	1	1	99.9			
ASC parameters						
Automatic dilution re-measurement	ON					
Blank Measurement alternative	OFF					
1) First reagent position	R1					
2) Second reagent position	R2					
3) Third reagent position						
4) Fourth reagent position						
Mixing	ON					
Blended sample injection volume	50 µl					
Inspection speed	25 µl/sec					
Suction discharge times	5 times					
Suction speed	130 µl/sec					
Discharge speed	330 µl/sec					
STD						
Concentration	Standard sample position	Sample amount	R1 amount	R2 amount	Total	
No	ppm	R1 to R8/1 to 60	(µl)	(µl)	(µl)	
1	0.0000	R3	0	495	5	
2	0.5000	R3	50	445	5	
3	1.0000	R3	100	395	5	
4	1.5000	R3	150	345	5	
5	2.0000	R3	200	295	5	
		Sample position	Sample amount	R1 amount	R2 amount	Total
		R1 to R8/1 to 60	(µl)	(µl)	(µl)	(µl)
Blank		R1	500	0	0	500
Drift						****
Unknown sample			5	495	0	500

Fig. 6 Conditions for Mn

***** Flame quantitative measurement (single drop method) ***** 1991/12/27 12:57:17 ***

Element name	Mg
Wavelength	285.2 nm
Method of concentration calculation	Calibration curve method
Analyzed by	
Remarks	

SAMPLE NO.	SAMPLE NAME	CONC. (ppm)	ABS. (279.5nm)	WEIGHT (g)	VOLUME (ml)	DILUTION RATIO	ACTUAL CONC.	FACTOR
1	GK-12	0.8846	0.0942	1.00	100.00	100.00	0.88 (x)	1.00
2	GK-16	1.8363	0.1923	1.00	100.00	100.00	1.84 (x)	1.00

Fig. 7 Analytical Results for Mn



SHIMADZU CORPORATION, International Marketing Division

3, Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101, Japan Phone:81(3)3219-5641 Fax:81(3)3219-5710

Cable Add.:SHIMADZU TOKYO Overseas Telex No.0232-3291 (SHMDT J)